

# REMOVAL OF COLOR AND COD FROM C.I. ACID RED 52 AQUEOUS SOLUTION BY NaOCl AND H<sub>2</sub>O<sub>2</sub> OXIDATION PROCESSES

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## ABSTRACT

*Removal of color from aqueous solution of C.I. Acid Red 52, a xanthene dye was investigated by chemical coagulation with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, AlCl<sub>3</sub>, FeSO<sub>4</sub>, FeCl<sub>3</sub> and advanced oxidation process (AOP) employing NaOCl and H<sub>2</sub>O<sub>2</sub>. Coagulation experiments yielded no color removal whereas studies with AOP resulted in good color removal. Oxidation with NaOCl produced a color removal of 87% and a COD removal of 53% at a pH of 9.5 whereas H<sub>2</sub>O<sub>2</sub> exhibited a color removal of 98% and COD removal of 67% at a pH of 12. Color removal from the dye molecule subjected to oxidation with oxidants NaOCl and H<sub>2</sub>O<sub>2</sub> may be delineated through step wise oxidative degradation of the chromophoric groups in the dye molecule to colorless hydroxyl, carbonyl, ozonide, carboxylic acids residues and finally to CO<sub>2</sub> and H<sub>2</sub>O depending on the nature, concentration and contact time of the oxidant used and also on the pH of the medium. A probable mechanism of chemical degradation of the dye by AOP is also proposed.*

**Key words:** Acid Red 52, Chemical Oxidants, Degradation Paths, Color and COD Removal.

**Cite this Article:** K. Thasilu and J. Karthikeyan, Removal of Color and COD from C.I. Acid Red 52 Aqueous Solution by NaOCl and H<sub>2</sub>O<sub>2</sub> Oxidation Processes, *International Journal of Civil Engineering and Technology*, 7(1), 2016, pp. 47-59.

<http://www.iaeme.com/IJCET/issues.asp?JType=IJCET&VType=7&IType=1>

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## 1. INTRODUCTION

Textile dye effluents are intensively colored due to the presence of residual dyes. Discharge of colored effluents into waters imparts color and impairs their intended beneficial use. They are toxic and aesthetically displeasing. Varieties of dyes and their steadfastness renders decoloration of synthetic dye effluents rather difficult and challenging task for the environmental engineers.

A number of physicochemical, biological and chemical processes have been investigated for removal of color from synthetic colored effluents. Coagulation and flocculation using Fe, Al and Mg salts has been investigated for decoloration of dye bearing wastewaters [1], [2], [3], [4] and [5]. Limitations of coagulation and flocculation to produce good quality effluent, besides sludge handling problems have rendered the process less attractive. Adsorption onto activated carbon is an effective method [6] and [7]. But the high cost of activated carbon prompted many investigators to explore a wide variety of low cost materials like fly ash, peat, sawdust, brown coal and bagasse pith for color removal [7] and [8]. BOD, COD and organic substances are removed by activated sludge, aerated lagoons or trickling filters [9]. However, biological processes are ambiguous, unreliable and are seldom capable of achieving desired degree of color removal [2], [10] and [11]. Dissolved salts and metal ions are removed by reverse osmosis or electrodialysis [12]. Technical and economical constraints of membrane separation processes like reverse osmosis [13], [14], [15] and [16] make the process less attractive. Conventional primary and secondary treatment methods are ineffective for color removal due to the complex structure and stability of dyes and produce large amount of sludge the disposal of which need further treatment [2] and [17].

From these perspectives, advanced oxidation process (AOP) appears to be a potential alternative and is highly efficient for decolorisation and degradation of textile dye effluents. Oxidation is defined as the transfer of one or more electrons from an electron donor (reducant) to an electron acceptor (oxidant) which has a higher affinity for electrons. These electron transfers result in the chemical transformation of both the oxidant and the reducant, producing in some cases chemical species with an odd number of valence electrons. These species, known as free radicals, are highly unstable and reactive because of the unpaired electrons. Oxidation reactions that produce radicals tend to be followed by additional oxidation reactions between the radical oxidants and other reactants (both organic and inorganic) until thermodynamically stable oxidation products are formed. The ability of an oxidant to initiate chemical reactions is measured in terms of its oxidation potential. Hydroxyl radicals ( $\text{HO}\cdot$ ), ozone, and chlorine are powerful oxidants with oxidation potentials of 2.85, 2.07 and 1.49 electron volts respectively [18]. Several technologies such as Fenton, photo-Fenton, ozonation, photocatalysis, etc. are included in the group of the AOP and the main difference between them is the source of radical production [19].

Sodium hypochlorite degrades the organic dyes effectively [20]. The dye containing amino or substituted amino and sulphonic acid groups on the naphthalene ring are most susceptible for chlorine decolorisation [21]. The rate determining step in the oxidation degradation of azo dyes was the cleavage of azo linkage moiety [22]. Investigated color removal (45%) from textile wastewaters and reported that  $\text{H}_2\text{O}_2$  alone has been found less effective [23]. Investigations on textile effluents [24] revealed that efficiency of  $\text{H}_2\text{O}_2$  alone is very much less (21%).

In the present study, decolorisation of a xanthenes dye C.I. Acid Red 52 was investigated by coagulation process employing  $\text{FeCl}_3$ ,  $\text{Al}_2(\text{SO}_4)_3$ ,  $\text{AlCl}_3$  and  $\text{FeSO}_4$ . Further color removal was also investigated by AOP using oxidizing agents like  $\text{NaOCl}$  and  $\text{H}_2\text{O}_2$ . Efficiency of color removal was investigated in terms of percent color removal by changing oxidant dose, contact time and solution pH. At optimum conditions for color removal, COD removal data was also obtained.

## 2. EXPERIMENTAL

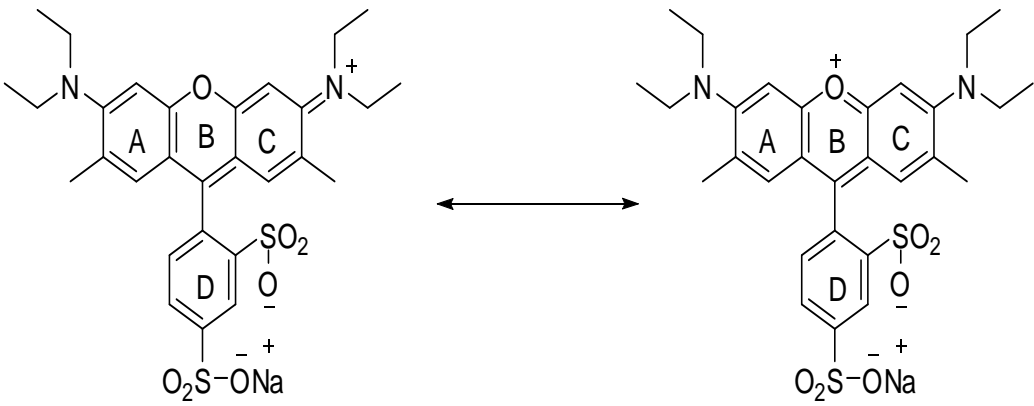
### 2.1. Chemicals

All chemicals used in the experimental work were of Analytical Reagent grade. FeCl<sub>3</sub>, Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, AlCl<sub>3</sub>, NaOCl (4% W/V available chlorine, Merck), H<sub>2</sub>O<sub>2</sub> (30% V/V, Merck), FeSO<sub>4</sub> (Merck, GR) were used in the present study. For pH adjustment, 0.1N H<sub>2</sub>SO<sub>4</sub> and 0.1N NaOH were used.

### 2.2. C.I. Acid Red 52 dye

C.I. Acid Red 52 (Atul industries, Ahmadabad, Gujarat, India), a textile dye was employed as test dye in the present study. Some important properties of this acid dye are presented in Table 1.

**Table 1** Important Properties of C.I. Acid Red 52

Dye/Properties	
Chemical name and structure: Sodium 4-(6-(diethylamino)-3-(diethyliminio)-3H-Xanthen-9-yl) benzene-1,3-disulfonate	
	
<p style="text-align: center;">Iminium form; Red in color                      Oxonium form; Deep red in color</p>	
C.I. Number	45100
CAS No	3520-42-1
Chemical Class	Xanthene
Molecular formula	C <sub>27</sub> H <sub>29</sub> N <sub>2</sub> NaO <sub>7</sub> S <sub>2</sub>
Molecular weight, g/mol	580.65
Maximum Wave length (nm)	570

## 2.3. Experimental Methodology

### 2.3.1 Dye stock solution

Appropriate amount of accurately weighed dye was dissolved directly in distilled water to prepare test dye solution of  $100\text{mgL}^{-1}$  as and when required.

### 2.3.2. Color and COD Measurement

Dye color concentrations were determined colorimetrically using a VIS Spectrophotometer (Spectrophotometer 106, Systronics India Limited, Hyderabad, India) by measuring %T/OD at optimum wavelength and reading for the dye color concentration from the calibration curve. To evaluate color removal, residual color concentration after the experiment was measured. Similarly, residual COD was measured by standard methods as per APHA.

### 2.3.3. Coagulation experiments

Coagulation experiments were conducted by standard jar test procedures using a six place jar\_test apparatus (Biological Enterprises, Delhi, India). To a 300 mL sample of the dye stock solution taken in a beaker, coagulant dose in the range of  $1\text{gL}^{-1}$  to  $16\text{gL}^{-1}$  was added and mixed for a typical rapid mixing time of 2 minutes at 120 rpm followed by slow mixing at 30rpm for 20 minutes. The contents were then sedimented for 2 hours and the supernatant aliquot was withdrawn and analyzed for color concentration.

### 2.3.4. Advanced Oxidation Process (AOP) Methods

Color removal by AOP was investigated employing oxidants like NaOCl and  $\text{H}_2\text{O}_2$ .

### 2.3.5. Test procedure with Sodium hypochlorite, Hydrogen peroxide

To a 300 mL of test dye stock solution taken in a 1000 mL glass beaker, varying doses of oxidants like NaOCl or  $\text{H}_2\text{O}_2$  was added and the contents were mixed, first at high speed (100-120 rpm) for one minute and then speed was reduced to 30-40 rpm and mixed for 20 minutes and equilibrated for two hours and analysed for residual color concentration.

Experimentation was conducted in three stages. In the first stage, varying doses of NaOCl from  $0.67\text{gL}^{-1}$  to  $8\text{gL}^{-1}$  and  $\text{H}_2\text{O}_2$  doses from  $5\text{gL}^{-1}$  to  $60\text{gL}^{-1}$  were added, residual color concentration was measured and least chemical dose producing maximum color removal was designated as favorable dose. In the second stage 40-80% of favorable dose was added to the test dye stock solution maintained at a pH of 3, 4, 5, 6, 8, 9, 10.5 and 12; reaction carried out and the pH value producing maximum color removal was designated as favorable pH. In stage three, experiments were conducted at favorable pH; employing varying doses just in the range of favorable dose and optimum dose i.e. the least dose that produces maximum color removal was determined.

## 3. RESULTS AND DISCUSSION

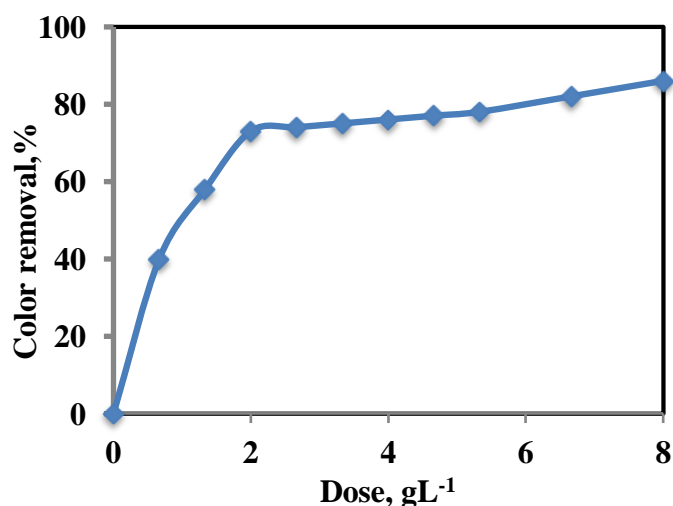
All experimental results are presented in graphical form and are followed by a discussion of the results.

### 3.1. Color removal by chemical coagulation

Chemical coagulation with Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>, AlCl<sub>3</sub>, FeSO<sub>4</sub> and FeCl<sub>3</sub> has not resulted in any color removal. A poor color removal with inorganic coagulants like Alum and Ferrous sulphate for amphoteric leveling type acid dye C.I. Acid Red 52 which is in tune with the present observation [2] and [25].

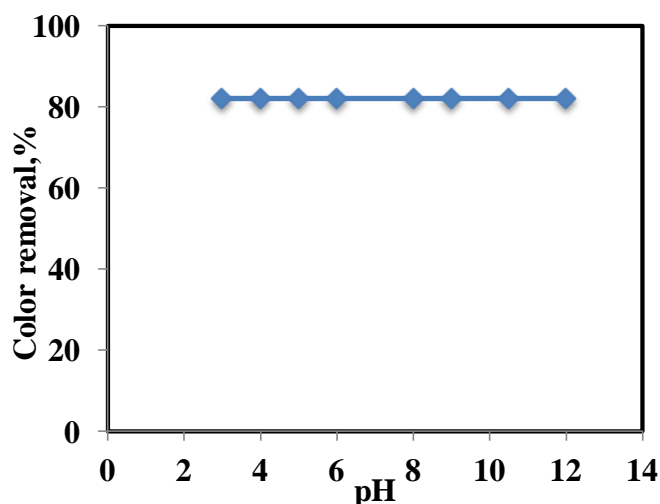
### 3.2. Color removal by sodium hypochlorite

When NaOCl of 4% was added in the dose range of 0.67g L<sup>-1</sup> to 8g L<sup>-1</sup>, color removal increased rapidly from 40 to 76% at 2g L<sup>-1</sup> and slowly to 86% at 8 g L<sup>-1</sup> and the results are presented graphically in Fig.1.



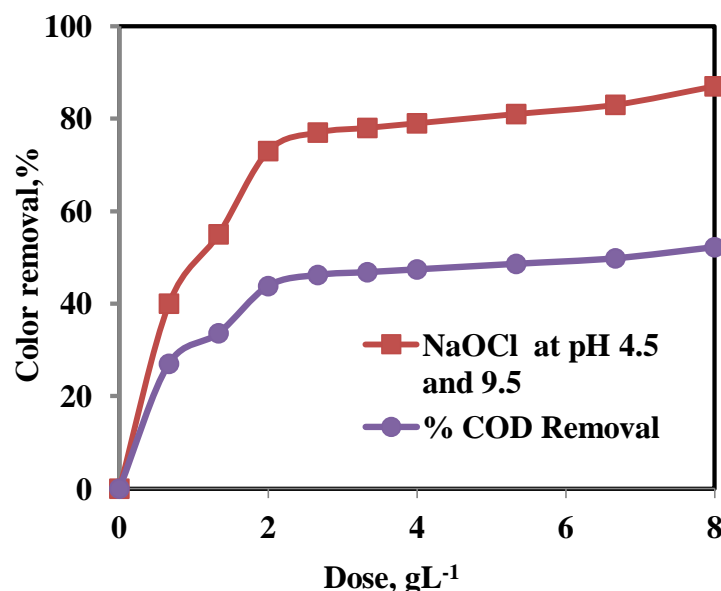
**Figure 1** Color removal at varying doses of NaOCl

As the color removal exhibited an increasing trend, a dose of 6.67g L<sup>-1</sup> was considered as favorable dose and color removal was investigated at different pH values and the results are portrayed in Fig. 2. It follows from the color removal data presented in Fig. 2 that pH of the test dye solution does not have any influence on color removal and color removal remained fairly constant at around 82%



**Figure 2** Effect of pH on color removal at favorable dose

Color removal was investigated at both acidic pH 4.5 and alkaline pH 9.5 in the dose range of  $0.67\text{gL}^{-1}$  to  $8\text{gL}^{-1}$  and the results are shown in Fig. 3. It may be noted from the color removal data presented in Fig. 3, that color removal follows similar trend with a maximum removal of 87% at  $8\text{gL}^{-1}$  and that there is no change in color removal at acidic or alkaline pH at all dosages. Removal of COD was 24% at a dose of  $0.67\text{gL}^{-1}$  which increased to 52% at a dose of  $8\text{gL}^{-1}$ .



**Figure 3** Color and COD removal at varying doses of NaOCl at pH 4.5 and 9.5

Chlorine is the active ingredient in NaOCl and its usage and chemistry in water is well understood. In a diluted NaOCl solution, over pH range of 5-10 there exists equilibrium between undissociated HOCl and dissociated  $\text{OCl}^-$  ion. While HOCl is useful as an effective disinfection agent [26] and widely used in water treatment, the  $\text{OCl}^-$  finds wide application in wastewater treatment as an oxidizing agent [27]. While designing chlorine fast reactive dyes observed that dye containing amino, substituted amino and sulphonic acid groups on the naphthalene ring are most susceptible and treatment with NaOCl results in an attack of the amino group of the dye molecule by  $\text{Cl}^+$ , initiating and accelerating cleavage of azo linkages and increasing concentration of chlorine favors decolorisation process [21].

It was reported that NaOCl degrades the organic dyes effectively owing to its strong oxidative capacity [20]. Rate of color reduction of azo dye Orange II by NaOCl in aqueous solution was observed to be dependent on pH and increased upto a pH of 9.3 and reported competitive inhibition at  $\text{pH} > 10$  leading to a decrease in color removal [28].



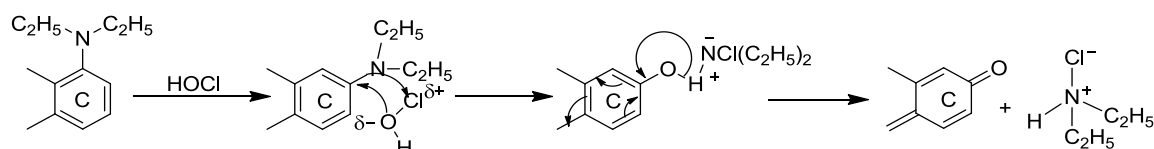
The rate determining step in the oxidation degradation of azo dyes was the cleavage of azo linkage moiety by  $\text{OCl}^-$  [22]. The Cl atom in  $\text{OCl}^-$  behaves as  $\text{Cl}^+$ , a strong electrophile, that combines with a pair of electrons in the C-N parts of the substrate that has high electron densities. The azo linkage of the dye has a high electron density and hence is readily chlorinated by  $\text{OCl}^-$  having the high electrophilicity. The electrophilic action of  $\text{OCl}^-$  towards nucleophilic N atoms in the molecule is responsible for the decolorisation [29].

Investigations on treatment of simulated wastewater containing Reactive Red 195 by zero-valent iron/activated carbon (ZVI/AC) combined with microwave discharge

electrode less lamp/sodium hypochlorite (MDEL/NaOCl) and reported the synergistic effect of ZVI/AC and MDEL/NaOCl resulted in breaking the azo bond firstly and then degrading the aromatic amine products finally to CO<sub>2</sub> and H<sub>2</sub>O and reported that acidic or neutral conditions were beneficial for dye degradation [30]. Photocatalytic degradation of Reactive Red 195 in aqueous solution using UV, NaOCl and TiO<sub>2</sub> Sep/nano particles indicated a maximum color removal of 99.90% at a dye concentration of 250 mg/L with 50.37 mM of NaOCl, 0.1 g/L of TiO<sub>2</sub> + Sep and pH of 5.45 in 3 hr [31].

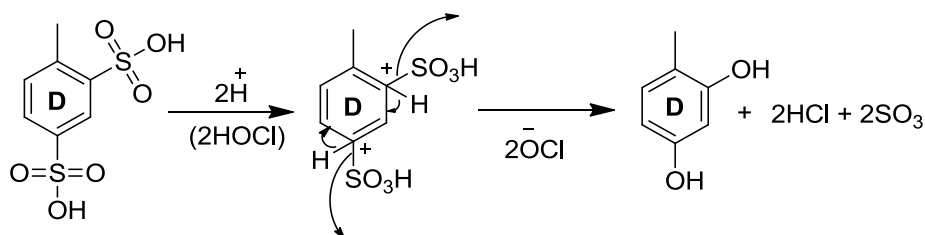
### 3.2.1. Mechanism of decolorisation of the dye compound with NaOCl

The dye molecule consists of four aromatic rings substituted with amino acid sulphonate groups. When NaOCl is added to the aqueous dye solution, NaOCl dissociates and forms HOCl. Because of large difference in the electrochemical nature of oxygen and chlorine, the oxygen being more electronegative than chlorine, HOCl exists in ionic form as HO<sup>-</sup>Cl<sup>+</sup> in water [32]. The Cl<sup>+</sup> of HOCl reacts with dye molecule and this results in the cleavage of C<sub>ar</sub>-N azo bond of the C<sub>ar</sub>-N(CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub> chromophore which facilitates OH bonding at the electrophilic carbon atom of the C<sub>ar</sub>-N(CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub> [21]. This reaction may lead to the formation of corresponding quinones, through the phenolic intermediate compound with the elimination of water soluble quaternary diethyl ammonium chloride as outlined in Scheme 1. Similar reaction occurs at the other C<sub>ar</sub>-N(CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub> group present in the ring A of the dye compound.

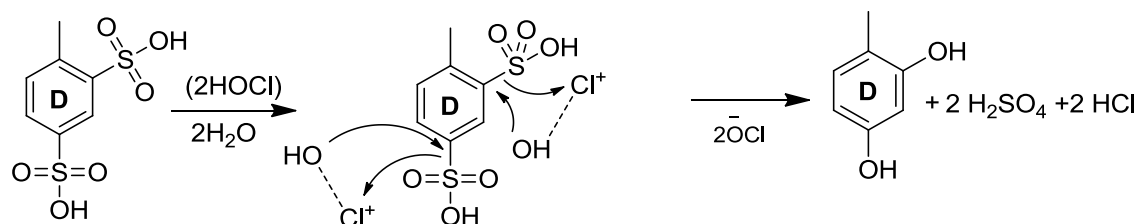


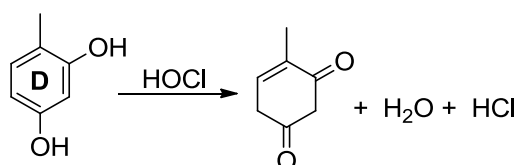
**Scheme 1** Oxidative degradation of C<sub>ar</sub>-N(CH<sub>2</sub>CH<sub>3</sub>)<sub>2</sub>

The C<sub>ar</sub>-SO<sub>3</sub><sup>-</sup> present in the ring D of the dye may undergo hydro-de-sulfonation reaction during the process of its decolorisation. The HOCl released during oxidative treatment with NaOCl converts it to an easily oxidisable colorless phenolic compound [34] as presented in Scheme 2.

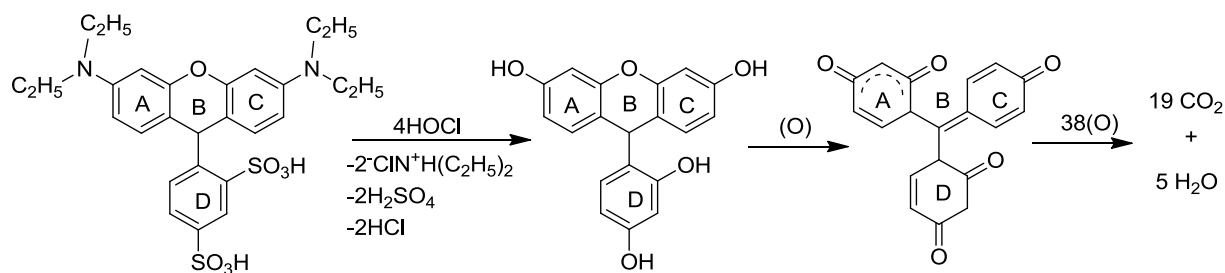


Alternatively the C<sub>ar</sub>-SO<sub>3</sub>H may get degraded by oxidation of the C<sub>ar</sub>-S bond by HOCl with hydroxylation of C<sub>ar</sub> atom.



**Scheme 2** Conversion of Car-SO<sub>3</sub>H to Car-OH and Car=O

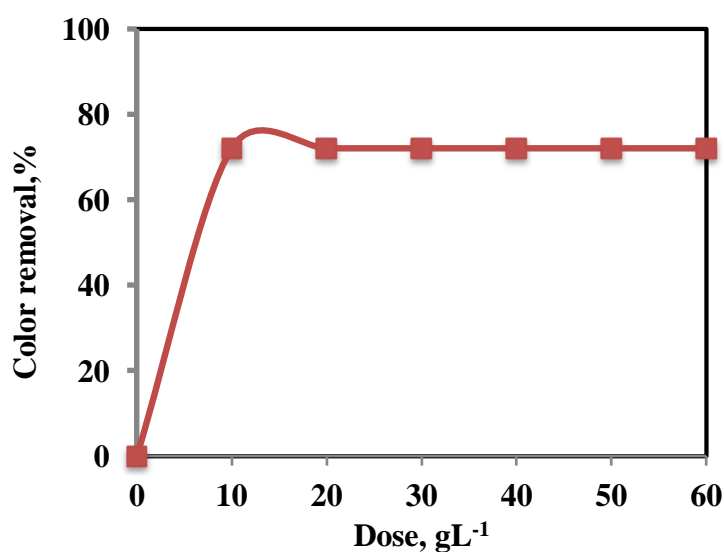
The ether functional group connecting the ring A and C in the dye being comparatively unreactive may not initially degrade during oxidative decolorisation of the dye present in water by HOCl. But after prolonged exposure to HOCl, this group may also get cleaved and lead to the total oxidation of the dye to CO<sub>2</sub> and H<sub>2</sub>O through corresponding hydroxy and quinone compounds depending on the contact time, concentration of the oxidant with the dye molecule as shown in Scheme 3.

**Scheme 3** Degradation of Car-O-Car in the dye

### 3.3. Color removal by Hydrogen Peroxide

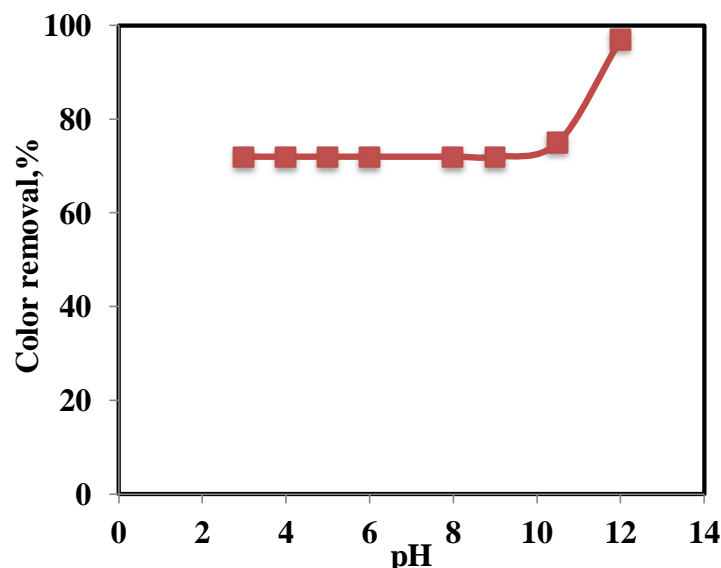
H<sub>2</sub>O<sub>2</sub> is a dibasic acid and acts as strong oxidant since it could release highly reactive nascent oxygen in acidic and alkaline solutions [33]. Hydrogen peroxide oxidizes aqueous organic residues effectively with atom efficiency of 47% with the formation of H<sub>2</sub>O as a byproduct [35] and is increasingly used as a green oxidant in wastewater treatment.

In the present study, H<sub>2</sub>O<sub>2</sub> was added in doses from 5 gL<sup>-1</sup> to 60 gL<sup>-1</sup> and the results of color removal are presented in Fig.4

**Figure 4** Color removal at varying doses of H<sub>2</sub>O<sub>2</sub>

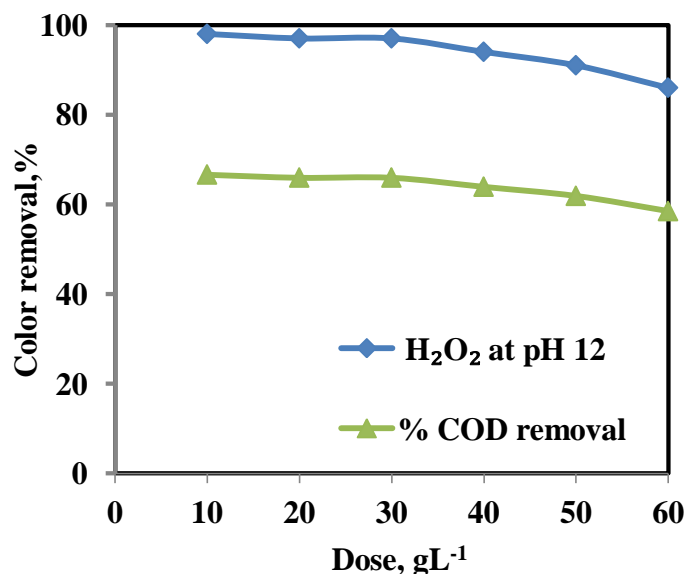


It follows from the results presented in Fig.4 that color removal increased rapidly to 72% at a dose of 10 gL<sup>-1</sup> H<sub>2</sub>O<sub>2</sub> dosage and remained constant thereafter upto a dosage of 60 gL<sup>-1</sup>. A dose of 30 gL<sup>-1</sup> was selected as the test dose and oxidation experiments were conducted to find the effect of pH of the dye solution on the extent of color removal at different initial pH values ranging from 3.0 to 12.0 and color removal results are presented in Fig.5.



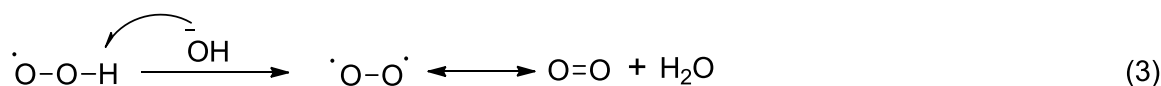
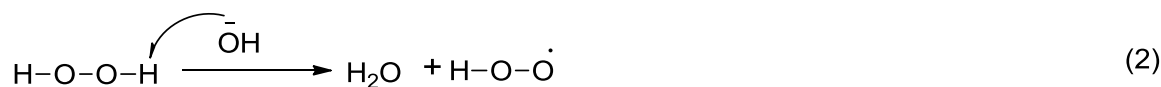
**Figure 5** Effect of pH on color removal at favorable dose

It may be observed from the Fig. 5 that there was no change in % color removal as the pH was increased from 3.0 to 9.0 (72%), but color removal increased slightly at pH 10.5 to 75% and suddenly increased to 97% as pH was increased to 12.0. A system pH of 12.0 was selected as favorable pH and experiments were conducted at favorable pH of 12.0 employing varying doses from 5g/L to 60 gL<sup>-1</sup> and the results are presented in Fig. 6. It may be inferred from color removal data presented in a Fig. 6 that favorable pH not only results in a reduction in the dosage of H<sub>2</sub>O<sub>2</sub> (10 gL<sup>-1</sup>), but also results in increased color removal (98%). A maximum COD removal of 67% was obtained at a dosage of 10gL<sup>-1</sup>.



**Figure 6** Color and COD removal at varying doses of H<sub>2</sub>O<sub>2</sub> at favorable pH

Increased removal at alkaline pH may be attributed to the generation of highly reactive peroxide ( $\text{HOO}^\bullet$ ) free radical and dioxygen biradical from the  $\text{H}_2\text{O}_2$  under basic conditions as shown by the following process.

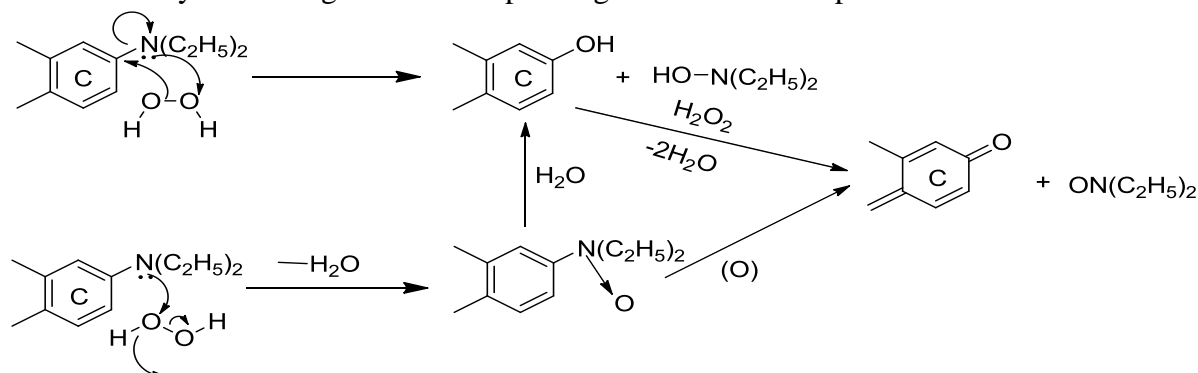


Little information is available on the use of  $\text{H}_2\text{O}_2$  for color removal from textile dye wastes. Color removal from textile wastewaters by  $\text{H}_2\text{O}_2$  alone has been found ineffective at both acidic and alkaline pH conditions [23]. It was observed that the efficiency of color removal by  $\text{H}_2\text{O}_2$  alone was negligible and reported a maximum color and COD removal of 21% and 32% for blue and 21% and 44% for black textile dye effluent respectively. In the presence of catalyst like ozone or UV or Fenton, hydroxyl radicals react with organic matter and decompose it leading to mineralisation of its constituent elements [24].

### 3.3.1. Dye degradation pathway by $\text{H}_2\text{O}_2$

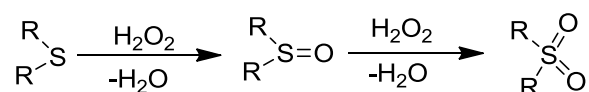
Decolorisation of the dye molecule involves oxidative destruction of the chromophoric tertiary amine and sulphonic acid functional groups of the dye by Hydrogen peroxide. They are likely to be converted into corresponding N-oxide and hydroxy derivatives first and subsequently to colorless small organic molecular residues. The reactions involved during this oxidative decolorisation process may be depicted in the following schemes.

Aromatic amines are known to be directly oxidized to hydro quinones and quinones by atmospheric oxidation [36]. In the same way, the tertiary amino group present in the dye is converted to the corresponding hydroxyl compound and quinone either directly or through the corresponding N-oxides as depicted in Scheme 4.



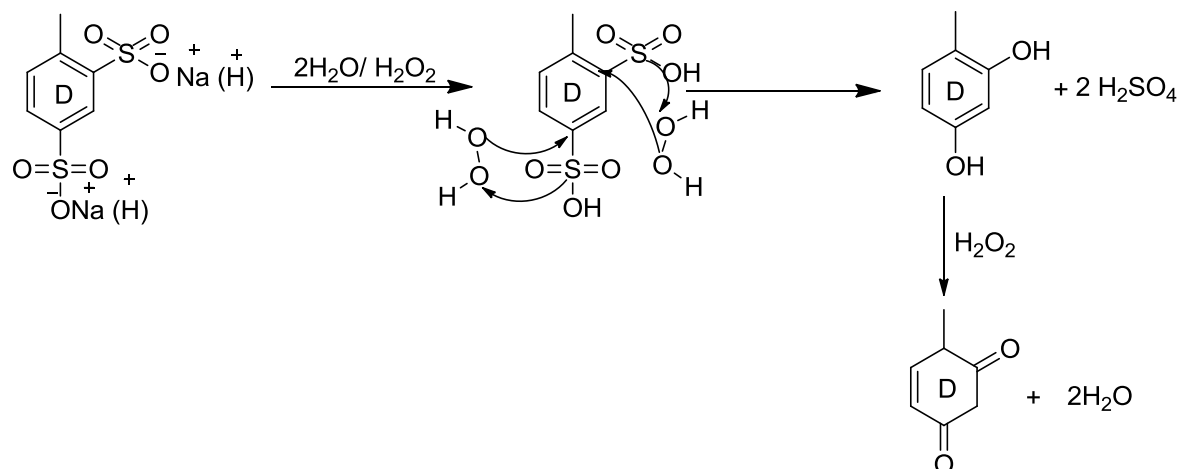
**Scheme 4** Hydrogen peroxide oxidation of Car-N ( $\text{C}_2\text{H}_5$ )<sub>2</sub>

Organic sulfides are oxidized by  $\text{H}_2\text{O}_2$  progressively to sulfoxides and sulfones depending on its concentration as portrayed in Scheme 5.



**Scheme 5** Oxidation of C-S-C functional group

Likewise oxidation of sulfonic acid  $-(C-SO_2-OH^-)$  or its sodium salt  $(C-SO_2-O^- Na^+)$  present in the dye molecule may transfigure to sulfuric acid and corresponding hydroxyl and carbonyl compounds as described in Scheme 6.



**Scheme 6** H<sub>2</sub>O<sub>2</sub> oxidation of Car- SO<sub>3</sub>OH group

The central xanthene ring (B) in the dye being aromatic may remain intact without undergoing cleavage of its C-O-C ether group under these experimental conditions.

#### 4. CONCLUSION

Decolorisation and degradation of Acid Red 52 was investigated by chemical coagulation and advanced oxidation process (AOP). Coagulation experiments yielded no color removal whereas studies with AOP resulted in good color removal. Oxidation with NaOCl produced a color removal of 87% and COD removal of 53% at a pH of 9.5 and no significant change in color removal was noticed at acidic and alkaline pH. Treatment with H<sub>2</sub>O<sub>2</sub> exhibited a good color removal of 98% and COD removal of 67% at a pH of 3 with dosage of 10g/L<sup>-1</sup>. The order of oxidants in color and COD removal was H<sub>2</sub>O<sub>2</sub>>NaOCl. A tentative scheme of mechanisms for the degradation of the dye by AOP is also proposed.

#### ACKNOWLEDGEMENTS

The authors acknowledge Prof. C. Devendranath Reddy (Retired), Department of Chemistry, S. V. University for his discussions in the chemistry part of this work.

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